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Naphtho[2,3-*c*]pyrrole-1,3(2*H*)-dione

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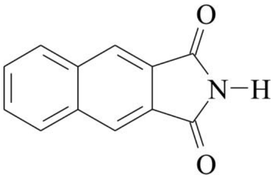
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.128; data-to-parameter ratio = 11.2.

The molecules of the title compound, 2,3-naphthalimide, $C_{12}H_7NO_2$, form an infinite hydrogen-bonded chain in the crystal structure parallel to the b axis. Within the chain, molecules linked by a nearly linear N—H···O interaction are related by a twofold screw axis.

Related literature

For related literature, see: Allen (2002); Bulgarovskaya *et al.* (1976); Janczak & Kubiak (1995); Janczak & Kubiak (2000); Janczak & Kubiak (2005); Pauling (1960).



Experimental

Crystal data

$C_{12}H_7NO_2$
M_r = 197.19
Monoclinic, $P2_1/n$
a = 8.158 (2) Å
b = 5.644 (1) Å
c = 19.034 (4) Å
β = 100.07 (3)°
V = 862.9 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 295 (2) K
0.42 × 0.34 × 0.28 mm

Data collection

Kuma KM-4 with CCD area-detector diffractometer
Absorption correction: analytical [face-indexed (*SHELXTL*; Sheldrick, 1990)]
T_{min} = 0.961, T_{max} = 0.974
4837 measured reflections
1869 independent reflections
1190 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.128
S = 1.01
1869 reflections
165 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O12^{i}	0.92 (2)	2.00 (2)	2.916 (2)	171 (2)

Symmetry code: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, -z-\frac{1}{2}$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2004); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED*, (Oxford Diffraction, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2064).

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supplementary materials

Acta Cryst. (2007). E**63**, o2627 [**doi:10.1107/S1600536807019022**]

Naphtho[2,3-*c*]pyrrole-1,3(2*H*)-dione

J. Janczak and **R. Kubiak**

Comment

The present study is a continuation of our investigations on the characterization of cyano compounds and the transformation of their cyano groups yielding various derivatives (Janczak & Kubiak, 1995; Janczak & Kubiak, 2000; Janczak & Kubiak, 2005). In order to expand the understanding of the functionality of the cyano groups, in this work we investigate the crystal structure of 2,3-naphthalimide, (I), obtained during hydrolysis of 2,3-dicyanonaphthalene. Depending on the hydrolysis conditions, the crystals of naphthalimide or pyrromellitic diimide are formed. The structure of pyrromellitic diimide is known and was described earlier (Bulgarovskaya *et al.*, 1976) while the crystal structure of 2,3-naphthalimide is reported here.

The molecule of (I) is almost planar, the deviation of the C, N and O atoms from the mean plane is smaller than 0.099 (2) Å (Fig. 1). The C—C bond lengths within the naphthalene ring exhibit a pattern of alternatively shorter and longer bonds that are typical for 2,3-substituted naphthalene derivatives (Allen, 2002).

The 2,3-naphthalimide molecules related by the twofold screw axis interact through N—H···O hydrogen bonds (Table 2) forming infinite chains parallel to the *b* axis (Fig. 2). The neighbouring chains, related by an iversion center, interact *via* π-π clouds of the aromatic rings, since the distance is comparable with the distance for interacting π-aromatic ring systems (Pauling, 1960). Both types of intractions are the key for the organization of the molecules in the crystal.

Experimental

The crystals of naphthalimide were obtained from 2,3-dicyanonaphthalene and H_2SeO_3 mixed together in the 1:2 molar ratio. The mixture was pressed into a pellet, inserted into an evacualted glass ampoule and then sealed. Next the ampoule was heated in the temperature gradient (hot zone at 150°C, cold zone at 100°C). After 4–5 h a colourless singe crystals suitable for the X-ray crystal analysis were formed at the cold zone. When the ampoule was heated at 180°C, the crystals of pyromellitic diimide were formed in the cold zone .

Refinement

The H atoms were loctated from difference Fourier maps and refined with the U_{iso}=1.2U_{eq} of the N or C atoms directly joined the H.

Figures

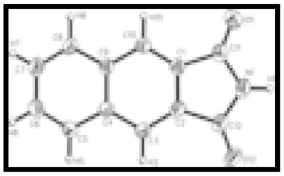

Fig. 1. A view of (I) showing displacement ellipsoids at the 50% probability level and H atoms as spheres of arbitrary radii. Hydrogen bonds are drawn as dashed lines.

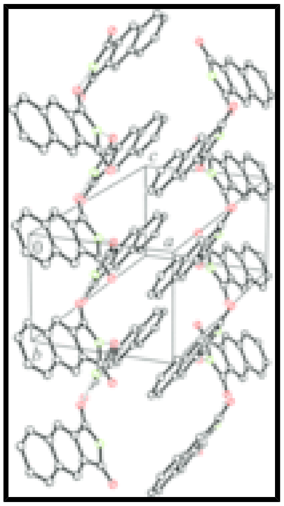

Fig. 2. A view of the crystal packing in (I) showing the hydrogen bonded chains along the *b* axis. Dashed line represents the hydrogen bonds. H atoms are omitted for clarity.

Naphtho[2,3-*c*]pyrrole-1,3(2*H*)-dione

Crystal data

$C_{12}H_7NO_2$

$M_r = 197.19$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 8.158\ (2)$ Å

$b = 5.644\ (1)$ Å

$c = 19.034\ (4)$ Å

$\beta = 100.07\ (3)$°

$V = 862.9\ (3)$ Å^3

$Z = 4$

$F_{000} = 408$

$D_x = 1.518$ Mg m^{-3}

$D_m = 1.51$ Mg m^{-3}

D_m measured by floatation

Mo *K*α radiation

$\lambda = 0.71073$ Å

Cell parameters from 853 reflections

$\theta = 2.2$–27.0°

$\mu = 0.11$ mm^{-1}

$T = 295\ (2)$ K

Paralellepiped, colourless

0.42 × 0.34 × 0.28 mm

Data collection

Kuma KM-4 with CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 1024x1024 with a blocks 2x2 pixels mm^{-1}

$T = 295(2)$ K

ω scans

Absorption correction: analytical [face-indexed (SHELXTL; Sheldrick, 1990)]

$T_{\min} = 0.961$, $T_{\max} = 0.974$

4837 measured reflections

1869 independent reflections

1190 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.026$

$\theta_{\max} = 27.1$°

$\theta_{\min} = 2.2$°

$h = -10 \rightarrow 10$

$k = 0 \rightarrow 7$

$l = -24 \rightarrow 2$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0781P)^2 + 0.0967P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.044$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.128$	$\Delta\rho_{max} = 0.32$ e Å^{-3}
$S = 1.01$	$\Delta\rho_{min} = -0.22$ e Å^{-3}
1869 reflections	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
165 parameters	Extinction coefficient: 0.018 (5)
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: difference Fourier map	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of F^2 > 2sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O11	0.5370 (2)	−0.4389 (3)	−0.13263 (8)	0.0508 (4)
O12	0.8153 (2)	0.2277 (3)	−0.18072 (7)	0.0501 (4)
N1	0.6682 (2)	−0.1191 (3)	−0.17438 (9)	0.0400 (4)
C1	0.6773 (2)	−0.1377 (3)	−0.05337 (9)	0.0314 (4)
C2	0.7594 (2)	0.0699 (3)	−0.06862 (9)	0.0322 (4)
C3	0.8314 (2)	0.2173 (4)	−0.01581 (10)	0.0353 (5)
C4	0.8256 (2)	0.1574 (3)	0.05576 (10)	0.0317 (4)
C5	0.9046 (3)	0.2966 (4)	0.11355 (11)	0.0395 (5)
C6	0.9046 (3)	0.2295 (4)	0.18195 (11)	0.0424 (5)
C7	0.8223 (3)	0.0219 (4)	0.19668 (10)	0.0420 (5)
C8	0.7422 (3)	−0.1142 (4)	0.14287 (10)	0.0377 (5)
C9	0.7428 (2)	−0.0541 (3)	0.07100 (9)	0.0299 (4)
C10	0.6657 (2)	−0.2002 (3)	0.01416 (10)	0.0330 (4)
C11	0.6158 (2)	−0.2583 (4)	−0.12208 (10)	0.0363 (5)
C12	0.7554 (2)	0.0801 (4)	−0.14671 (10)	0.0370 (5)

H1	0.661 (3)	−0.164 (5)	−0.2214 (12)	0.055 (7)*
H3	0.894 (3)	0.360 (5)	−0.0252 (13)	0.051 (6)*
H5	0.965 (3)	0.442 (5)	0.1041 (12)	0.058 (7)*
H6	0.967 (3)	0.319 (4)	0.2221 (12)	0.044 (6)*
H7	0.829 (2)	−0.025 (4)	0.2462 (11)	0.043 (6)*
H8	0.679 (3)	−0.255 (5)	0.1524 (13)	0.061 (7)*
H10	0.604 (3)	−0.341 (5)	0.0231 (12)	0.047 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O11	0.0642 (10)	0.0446 (9)	0.0412 (8)	−0.0103 (8)	0.0027 (7)	−0.0073 (7)
O12	0.0723 (10)	0.0486 (9)	0.0336 (7)	0.0009 (8)	0.0208 (7)	0.0094 (7)
N1	0.0509 (10)	0.0450 (10)	0.0242 (8)	0.0033 (8)	0.0066 (7)	−0.0023 (8)
C1	0.0367 (9)	0.0291 (9)	0.0285 (9)	0.0016 (8)	0.0063 (7)	−0.0019 (7)
C2	0.0406 (9)	0.0303 (9)	0.0272 (9)	0.0039 (8)	0.0099 (7)	0.0025 (7)
C3	0.0458 (10)	0.0287 (10)	0.0337 (10)	−0.0021 (9)	0.0132 (8)	0.0014 (8)
C4	0.0372 (9)	0.0280 (9)	0.0312 (9)	0.0007 (8)	0.0094 (7)	−0.0012 (7)
C5	0.0464 (11)	0.0351 (11)	0.0386 (11)	−0.0082 (9)	0.0118 (8)	−0.0066 (9)
C6	0.0479 (11)	0.0455 (12)	0.0335 (10)	−0.0035 (10)	0.0063 (8)	−0.0101 (9)
C7	0.0533 (11)	0.0465 (12)	0.0267 (9)	0.0018 (10)	0.0078 (8)	0.0010 (9)
C8	0.0478 (11)	0.0370 (11)	0.0298 (9)	−0.0019 (9)	0.0111 (8)	0.0029 (8)
C9	0.0355 (9)	0.0283 (9)	0.0267 (8)	0.0027 (8)	0.0079 (7)	−0.0003 (7)
C10	0.0410 (10)	0.0274 (10)	0.0312 (9)	−0.0017 (8)	0.0077 (8)	0.0003 (7)
C11	0.0410 (9)	0.0373 (11)	0.0296 (9)	0.0041 (9)	0.0034 (7)	−0.0038 (8)
C12	0.0445 (10)	0.0392 (11)	0.0282 (9)	0.0065 (9)	0.0092 (7)	0.0024 (8)

Geometric parameters (Å, °)

O11—C11	1.203 (2)	C4—C5	1.412 (3)
O12—C12	1.209 (2)	C4—C9	1.427 (3)
N1—C12	1.384 (3)	C5—C6	1.356 (3)
N1—C11	1.393 (3)	C5—H5	0.99 (3)
N1—H1	0.92 (2)	C6—C7	1.403 (3)
C1—C10	1.352 (3)	C6—H6	0.98 (2)
C1—C2	1.405 (3)	C7—C8	1.353 (3)
C1—C11	1.482 (3)	C7—H7	0.97 (2)
C2—C3	1.357 (3)	C8—C9	1.410 (2)
C2—C12	1.482 (2)	C8—H8	0.98 (3)
C3—C4	1.412 (3)	C9—C10	1.417 (3)
C3—H3	0.99 (3)	C10—H10	0.97 (3)
C12—N1—C11	112.83 (17)	C7—C6—H6	118.6 (13)
C12—N1—H1	122.3 (16)	C8—C7—C6	120.48 (18)
C11—N1—H1	124.2 (16)	C8—C7—H7	121.3 (13)
C10—C1—C2	121.94 (17)	C6—C7—H7	118.2 (13)
C10—C1—C11	130.55 (18)	C7—C8—C9	120.98 (19)
C2—C1—C11	107.51 (16)	C7—C8—H8	121.3 (14)
C3—C2—C1	121.24 (16)	C9—C8—H8	117.7 (15)

C3—C2—C12	130.52 (18)	C8—C9—C10	121.65 (17)
C1—C2—C12	108.19 (16)	C8—C9—C4	118.73 (17)
C2—C3—C4	118.99 (18)	C10—C9—C4	119.62 (16)
C2—C3—H3	122.5 (14)	C1—C10—C9	118.61 (18)
C4—C3—H3	118.3 (14)	C1—C10—H10	120.4 (14)
C3—C4—C5	122.05 (18)	C9—C10—H10	121.0 (14)
C3—C4—C9	119.56 (17)	O11—C11—N1	125.50 (18)
C5—C4—C9	118.35 (17)	O11—C11—C1	128.68 (19)
C6—C5—C4	121.05 (19)	N1—C11—C1	105.82 (17)
C6—C5—H5	119.3 (14)	O12—C12—N1	125.78 (19)
C4—C5—H5	119.6 (14)	O12—C12—C2	128.58 (19)
C5—C6—C7	120.38 (19)	N1—C12—C2	105.63 (17)
C5—C6—H6	120.9 (13)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O12^{i}	0.92 (2)	2.00 (2)	2.916 (2)	171 (2)

Symmetry codes: (i) −*x*+3/2, *y*−1/2, −*z*−1/2.

Fig. 1

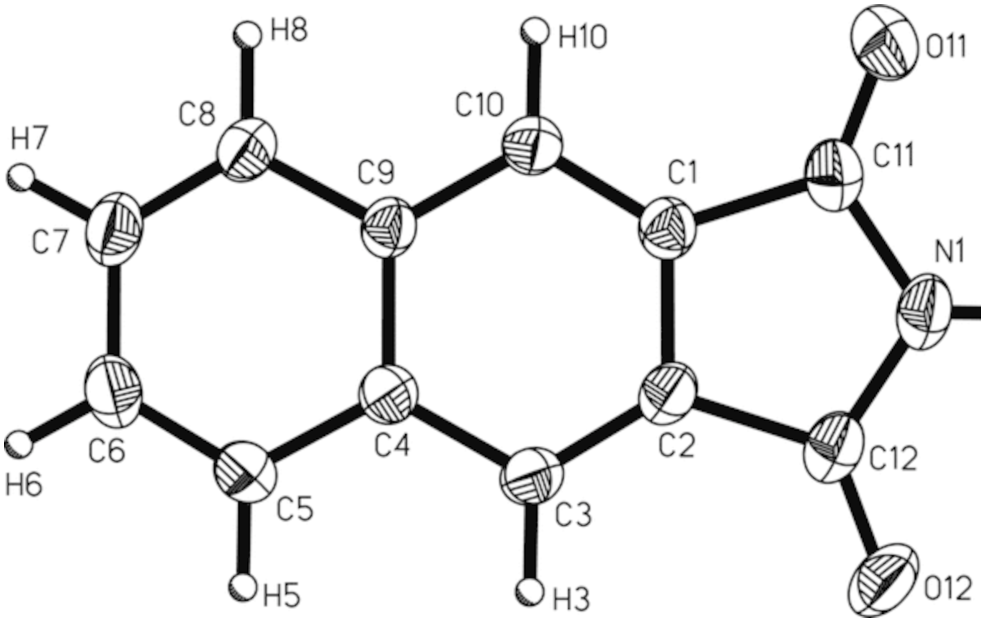
H8
H10
O11
H7
C8
C10
C11
C9
C1
C7
N1
H1
C6
C4
C2
C12
H6
C5
C3
H5
H3
O12

Fig. 2